# Effect of Electron-Beam Irradiation on Ethylene–Methyl Acrylate Copolymer

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**ABSTRACT:** Ethylene–methyl acrylate copolymer (Elvaloy 1330) was irradiated by an electron beam at different levels of radiation both in the presence and absence of a trimethylolpropane trimethacrylate sensitizer at various dosages of incorporation. The mechanical, thermal, and electrical properties of these samples were compared. The mechanical properties were observed to reach an optimum maximum around 6 Mrad of irradiation and 1 phr of sensitizer incorporation. Furthermore, an increase in either the radiation dose or the sensitizer level helped very little to further modify the properties. The thermal properties as determined by the thermogravimetric analysis and differential scanning calorimetry studies were quite supportive of the observation made during the study of the mechanical properties. The thermal stability of the irradiated samples underwent an increase with increasing electron-beam dosage. In a manner similar to those of the mechanical properties, the increase in thermal stability was found to reach a maximum at a particular level of treatment and sensitizer incorporation, beyond which there was marginal or no effect at all. The α transition temperature underwent a substantial increase with increasing crosslink density, as evidenced by the increase in gel content with increasing proportion of electron-beam radiation dose. The other glass-transition temperature, however, appeared to remain unaffected. The electrical properties, as described by the dielectric constant, volume resistivity, and breakdown voltage, appeared to be affected very little by the electron-beam radiation. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 112: 28–35, 2009

**Key words:** crosslinking; electron beam curing; electron beam irradiation; mechanical properties; thermal properties

## INTRODUCTION

Electron-beam irradiation is a potential method for the modification of polymers and compounds thereof. To achieve the optimum properties of polymers without any deterioration, the incorporation of some polyfunctional monomers that act as sensitizers helps to decrease the irradiation dosage level needed to attain the optimum number of crosslinks necessary for the properties desired. The polymers can, thus, be modified and crosslinked simultaneously without any processing difficulties.[1]

Radiation crosslinking thus endows polymers with special properties. The influence of electron-beam irradiation on the mechanical and dynamic mechanical properties, gel fraction, and crosslink density of terpolymeric fluorocarbon rubber was investigated by Banik and Bhowmick.[2] They found that with increasing radiation dose, the degree of crosslinking increased, which led to an increase in the modulus and glass-transition temperature, with a corresponding decrease in the elongation at break, set, and tan δ. The effect of a multifunctional monomer was realized only at a relatively higher level of trimethylolpropane trimethacrylate (TMPTMA), where an improvement in the strength and failure properties was observed. In another study,[3] radiation-crosslinked poly(vinyl chloride) was used as an insulating material for wires and cables. The limiting oxygen index was found to be as high as 39% with dioctyl phthalate (DOP) as a plasticizer and TMPTMA as a sensitizer. Ebe and Sasaki[4] described the electron-beam curing of aliphatic unsaturated polyesters for converting them to pressure-sensitive adhesives.[4] Bayram et al.[5] studied the effects of compatibilizing reactions on the viscoelastic properties and morphology of ethylene–methyl acrylate copolymers. Melt mixing was carried out in a batch mixer and in a corotating twin-screw extruder. The morphology of the reactive blends showed smaller domain sizes than the nonreactive blends, and the

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viscoelastic properties of the blends were very different. Mechanical testing of electron-beam irradiated low-density polyethylene/ethylene vinyl acetate (EVA) blends showed that the tensile strength of the samples increased with increasing irradiation dose up to 180 kGy, whereas the elongation at break decreased. The highest crosslink density was obtained at 20 wt % EVA. The gel content of low-density polyethylene/EVA blends increased with increasing irradiation dose, vinyl acetate, and EVA content.[6]

Ethylene–methyl acrylate copolymer is a thermally stable, polar polymer that produces no acidic or toxic byproducts when decomposed. It also possesses certain advantages over EVA and poly(vinyl chloride), which include superior thermal properties, flexibility without a plasticizer, low temperature flexibility, and others.[7] The high melt strength (low melt flow index), low temperature flexibility, polarity, increased flexibility with increased comonomer level, high filler acceptability, printability, and so on[8] further widen its fields of application. In this study, Elvaloy 1330 was selected as the base polymer, which was an ethylene copolymer containing 30% methyl acrylate and having a melt flow index of 3. It was subjected to varying dosages of irradiation at different sensitizer levels. The sensitizer used in this case was TMPTMA, and the results were analyzed with respect to the physicomechanical, electrical, thermal, and aging properties.

The principal objective of this study was to investigate the influence of crosslinking within this copolymer induced by electron beams at different doses both in the absence and in the presence of a suitable sensitizer at different levels on the various physical, mechanical, thermal, and electrical properties. The optimum doses of both irradiation and sensitizer were then traced.

## EXPERIMENTAL

### Materials

Elvaloy 1330 was supplied by M/s DuPont Specialty Elastomers (Mechelen, Belgium). An antioxidant, Irganox 1010 (hindered phenol type) was supplied by Ciba Specialty Chemicals (Basel, Switzerland), and TMPTMA was supplied from Sartomer Chemicals (Exton, PA).

### Preparation of the samples

Elvaloy 1330 (100 parts by weight) was mixed with Irganox 1010 (3 parts by weight) and a proportionate quantity of TMPTMA in a Haake Rheocord (Burladingen, Germany) instrument (model Haake Rheocord system 40) at a temperature of 80°C. Elvaloy was first allowed to melt in the mixer followed by the antioxidant and TMPTMA. The mix so obtained was sheeted out through an open mill with a 2 mm nip gap.

**TABLE I**
**Formulation of the Samples**

| Elvaloy 1330 (parts by weight) | TMPTMA (parts by weight) | Radiation dosage (Mrad) | Sample code |
|---|---|---|---|
| 100 | 0 | 0 | D000 |
| 100 | 0 | 3 | D003 |
| 100 | 0 | 6 | D006 |
| 100 | 0 | 9 | D009 |
| 100 | 0 | 12 | D012 |
| 100 | 0 | 15 | D015 |
| 100 | 1 | 0 | D100 |
| 100 | 1 | 3 | D103 |
| 100 | 1 | 6 | D106 |
| 100 | 1 | 9 | D109 |
| 100 | 1 | 12 | D112 |
| 100 | 1 | 15 | D115 |
| 100 | 2 | 0 | D200 |
| 100 | 2 | 3 | D203 |
| 100 | 2 | 6 | D206 |
| 100 | 2 | 9 | D209 |
| 100 | 2 | 12 | D212 |
| 100 | 2 | 15 | D215 |
| 100 | 3 | 0 | D300 |
| 100 | 3 | 3 | D303 |
| 100 | 3 | 6 | D306 |
| 100 | 3 | 9 | D309 |
| 100 | 3 | 12 | D312 |
| 100 | 3 | 15 | D315 |
| 100 | 4 | 0 | D400 |
| 100 | 4 | 3 | D403 |
| 100 | 4 | 6 | D406 |
| 100 | 4 | 9 | D409 |
| 100 | 4 | 12 | D412 |
| 100 | 4 | 15 | D415 |

The sheets were then compression-molded between aluminum foil at 110°C at a pressure of 140 kgf/cm$^2$ in an electrically heated press. Aluminum foil was used to reduce the shrink marks on the molded surface. The moldings were cooled under compression to maintain the overall dimensional stability of the samples.

### Radiation

The molded Elvaloy samples in the form of rectangular sheets were irradiated by an electron-beam accelerator at Nicco Corp., Ltd., Cable Division (Shyamnagar, India). Irradiation doses of 3, 6, 9, 12, and 15 Mrad were used. The formulation of the samples are given in Table I, and the specifications of the electron-beam accelerator are given in Table II.

### Characterization

#### Mechanical properties

*Tensile properties.* The tensile strength, elongation at break, and modulus at 100% elongation were measured on dumbbell specimens according to ASTM D

**TABLE II**
**Specifications of the Electron-Beam Accelerator**

| | |
|---|---|
| Energy range | 1–3 MeV |
| Beam power | 0.5–150 kW |
| Beam energy spread | 50–99.9% |
| Average current | 0.5–50 mA |
| Accelerating voltage frequency | 100 kHz–0.1 MHz |
| Duration | Any number of passes |
| Maximum pulse current | 50 mA |
| Minimum pulse current | 0.5 mA |
| Power supply voltage | 440 V |
| Power supply voltage frequency | 50 Hz |
| Consumption of power | 0.5–150 kW |
| Manufacturer | RDI |
| Model | 369/3 MeV/50 mA/220 mm Dynamitron oscillator model 375 |

412 in a Zwick-1445 (Burladingen, Germany) universal testing machine at a strain rate of 50 mm/min at 27 ± 2°C. An average was taken from four samples, and the experimental error was ±5%.

*Tear strength.* The tear strength was measured on trouser-shaped specimens following ASTM D 412 in a universal testing machine (Blue Star, Mumbai, India) (model ZMGI-25).

*Hardness.* The hardness of the prepared samples was measured by a Shore A durometer scale.

*Crosslink density.* We determined the crosslink density of the samples by measuring the gel content in a Soxhlet apparatus (S.C. Dey & Co., Kolkata, India) with ethyl acetate as a solvent. Absolutely dry samples were subjected to fractionation for 72 h at 65°C and dried in an air oven at 80°C until a constant weight was obtained.

The initial weight of the sample was taken as $W_1$, and the final weight was taken as $W_2$. The crosslink density was then given as $W_2/W_1 \times 100$.

### Thermal properties

*Thermogravimetric analysis (TGA).* Thermogravimetric measurements were carried out in a thermogravimetric analyzer (TA Instruments, New Castle, DE) (model TGA Q50 V6.1) within the temperature range 27–600°C at a heating rate of 10°C/min in an inert atmosphere of nitrogen.

*Differential scanning calorimetry (DSC).* The DSC measurements were carried out on a DSC apparatus (TA Instruments, New Castle, DE) (model DSC Q100 V8.1) within the temperature range −200 to 150°C. The heating rate was maintained at 10°C/min.

### Electrical properties

*Volume resistivity.* The volume resistivity of the samples were measured on a volume resistivity testing machine (model 4339B, Agilent Technology Japan, Ltd., Japan). Circular samples with an area of 19.64 $cm^2$ were used after they were cut from the molded sheets.

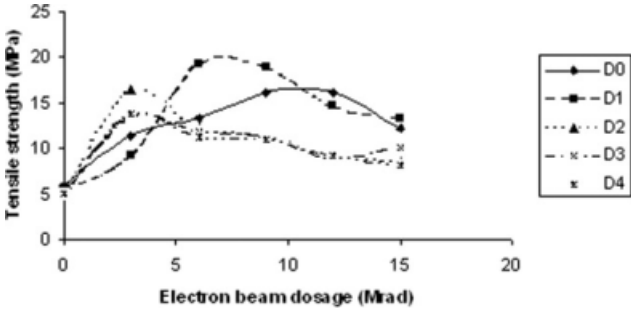



**Figure 1** Variation of the tensile strength with the electron-beam dosage.

*Dielectric constant.* Dielectric constants of the samples were measured in a dielectric constant meter from Sivananda Electric, Mumbai, India with an operating frequency of 1 MHz.

*Breakdown strength.* Breakdown strength was measured in a breakdown voltage (BDV) tester (Sivananda Electric, Mumbai, India) with a voltage application rate of 100–500 V/s.

## RESULTS AND DISCUSSION

### Mechanical properties

The variations in tensile strength, percentage elongation at break, and modulus at 100% elongation of the various samples under investigation are plotted against the irradiation dose in Figures 1–3, respectively. The tensile strength increased initially for all sensitizer levels (D006–D406), reached a maximum at different levels of irradiation for different doses of sensitizer incorporation, and gradually decrease in all of the samples under study. However, in the sample with 1-phr TMPTMA incorporation, a reduction in the tensile strength values was observed at a higher level of irradiation compared to other sensitized compounds as expected, and in the samples without any sensitizer, the optimum value of tensile strength was achieved at a still higher level.

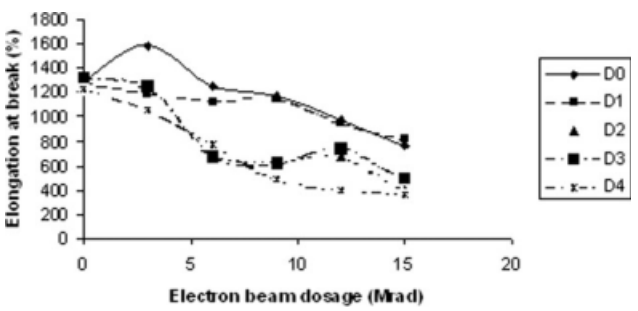



**Figure 2** Variation of the elongation at break with the electron-beam dosage.

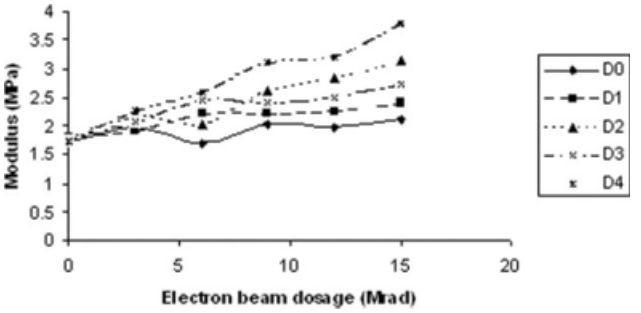



**Figure 3** Variation of the modulus at 100% elongation with the electron-beam dosage.

Interestingly, the influence of sensitizer was quite insignificant beyond 2 phr of its incorporation regardless of the dose of irradiation. Although in this study the sample with 1-phr TMPTMA as a sensitizer offered a maximum value of tensile strength at an approximately 6-Mrad irradiation dose, the other samples with higher sensitizer contents reached their corresponding peaks around 3 Mrad of electron-beam dosage. The sample without any sensitizer reached its peak around 12 Mrad, after which it deteriorated.

The initial rise in tensile strength for all of the samples (both sensitized and nonsensitized) may have been due to the formation of a network structure when the electron beam was incident over the samples. The process was made faster by the presence of a sensitizer, which accelerated the crosslinking process. The rise may also have been due to the crosslinks generated because of the electron-beam irradiation and the stress-induced orientation of the molecular chains in the direction of the applied mechanical force.[9]

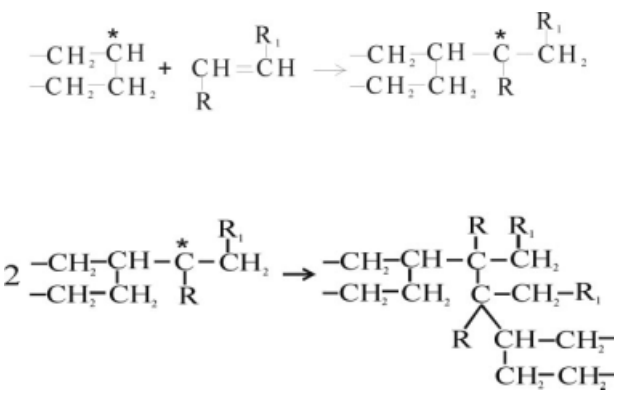
A high-energy electron beam usually reacts with materials by knocking off electrons and creating ions or free radicals. Sometimes, this is known as *ionizing radiation*. This ionizing radiation may cause a combination or coupling of two macroradicals and may sometimes lead to chain scission by termination through disproportionation, which leads to a deterioration in the mechanical properties. Which one of these two processes predominates over the other is measured by a *G* value. The *G* (free energy change) value depends on the susceptibility of the polymer to undergo crosslinking ($G_x$, free energy change during crosslinking) vis-à-vis degradation ($G_s$, free energy change during chain scission). The addition of a difunctional or trifunctional monomer (in our case, TMPTMA, a trifunctional monomer) to the curing system enhances gel formation because monomer endcapped macroradicals have a greater reactivity (as shown in Scheme 1).

The tensile strength was attributed to the crosslink density of the polymer. The sensitizer, TMPTMA, being trifunctional in nature, reacted with the system to attain a saturation level of crosslink density. This saturation level was attained faster in case of samples sensitized with 2 phr and greater values compared to the 1-phr sensitized ones because of the presence of more sensitizing material. As a result, the 2-, 3-, and 4-phr sensitized samples had optimized tensile values at a lower irradiation dosage compared to D106, where the optimization was reached at a higher level. At a similar maximum value of crosslink density, D206, D306, and D406 overcured, whereas D106 remained unabated and exhibited a maximum value at the same point of irradiation dosage.

The elongation at break (%) showed a gradual decreasing tendency over the entire range of irradiation dosage regardless of the presence or absence of sensitizer at any level. The gradual formation of more and more stiff, hard crosslinks within the polymer matrix with increasing dosage of irradiation (also exhibited in the curve of rising crosslink density) reduced the possibility of chain slippage and, consequently, the percentage elongation at break. At the higher doses of irradiation, when the crosslink density was high, the decoiling of the polymer chains was prevented, even on application of more load, which resulted in brittle fracture, which was depicted by the continuous fall in elongation over the entire range of irradiation.[10]

The effect of sensitizer and irradiation in the ethylene–methyl acrylate copolymer is quite prominently shown in Figure 3, which shows the modulus at 100% elongation of the samples under investigation as a function of irradiation dosage. As expected, the modulus steadily increased with increasing proportion of sensitizer and also as a function of irradiation dosage for any given level of sensitizer under study.

A continuous rise in the crosslink density (faster for the sensitized compounds) is quite evident in the plot of crosslink density versus irradiation dosage (Fig. 4). Interestingly, the differences in gel content



**Scheme 1** Crosslinking mechanism of the electron-beam technique with TMPTMA.

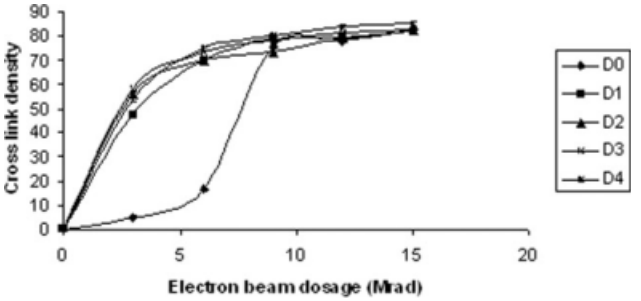



**Figure 4** Variation of the crosslink density with the electron-beam dosage.

over the range of sensitizer level under study were quite insignificant, and almost all of them leveled off along with the nonsensitized sample, particularly at higher irradiation dosages. We inferred that $G_s$ (chain scission) took over the $G_x$ (crosslinking process) at higher irradiation dosage once the optimum gel content (a measure of crosslink density) was reached, possibly because of an equilibrium reached between the rate of crosslink formation and the rate of chain scission.

The modulus and tensile strength of a cured sample is proportional to the number of crosslinks formed. As the radiation dose increased, the crosslink density (determined from gel content) increased, and hence, the modulus increased, as shown in Figure 3. The tensile strength is considered to be a function of crosslink density, energy dissipation, chain scission, hindrance to crystallization, and modification of the main chain, if any. We also observed that the stress-induced crystallization, as indicated from a sudden rise in stress, decreased with irradiation dosage, and consequently, the tensile strength deteriorated.[11] The tensile strength increased with increasing TMPTMA level and reached a maximum at 1 phr, which was an effect of crosslinking. The effect was most pronounced in the case of the 6-Mrad dose, after which the phenomenon of chain scission, rather than further crosslinking, influenced the mechanical properties. Beyond 1-phr TMPTMA,

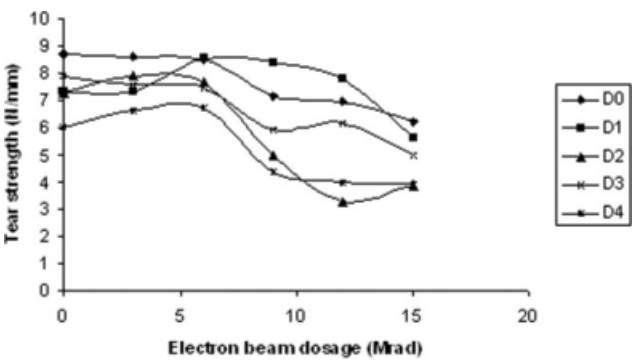



**Figure 5** Variation of the tear strength with the electron-beam dosage.

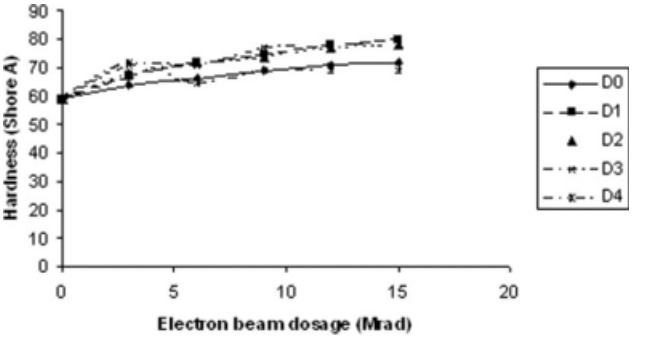



**Figure 6** Variation of the hardness with the electron-beam dosage.

molecular imperfection played an important role in the control of the morphology.[12]

The tear strength values, as shown in Figure 5, indicated a direct relationship to those of elongation at break. In this case also, the steady decrease with increasing irradiation dosage was a measure of increasing brittleness with increasing crosslink density.

The indentation hardness, a surface phenomenon, which reflects the resistance to local deformation, is a complex property related to modulus, tensile strength, elasticity, and plasticity. An inverse relationship between the depth of penetration (the lower the depth is, the higher the hardness is) of the indenter and the modulus was suggested by Nielsen[13]:

$$\mathrm{h} = [3/4(1 - v^2/\mathrm{E})]^{2/3}\mathrm{F}^{2/3}\mathrm{R}^{-1/3}$$

where $h$ is the depth of penetration, ν is Poisson's ratio, $E$ is the modulus, and $F$ is the total force on the spherical indenter of radius $R$.

In this case, as shown in Figure 6, the hardness increased marginally at all levels of TMPTMA incorporation and also increasing irradiation dose, regardless of sensitizer level. The different sensitizer-containing samples, however, differed very little over the range of irradiation under study.

## Thermal properties

### TGA

The thermogravimetric plots of Elvaloy 1330 and its series with 1-phr TMPTMA at different doses of irradiation are shown in Figure 7(a,b), respectively. It is quite interesting to note that the degradation temperature (i.e., the onset of degradation) of the samples under investigation rose consecutively as the dose of the electron beam increased. This also bore a direct proportionality with rise in crosslink density. As the crosslink density of the samples increased with increasing electron-beam dosage (Fig. 4), the ability to withstand thermal degradation also

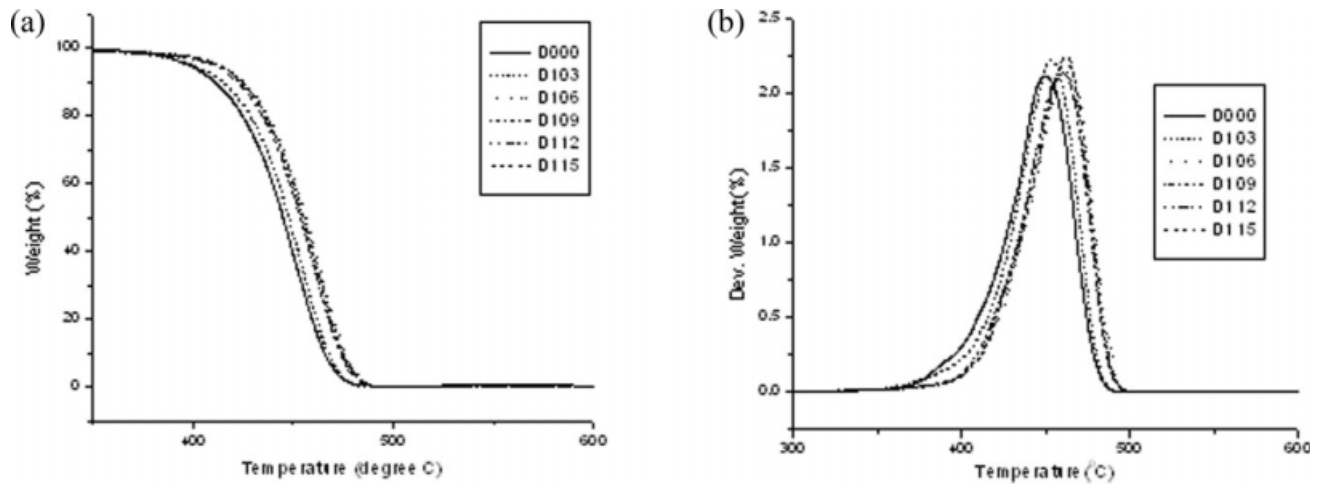



**Figure 7** (a) TGA curves at 1-phr TMPTMA incorporation. (b) Differential thermogravimetry curves at 1-phr TMPTMA incorporation.

increased because of the crosslinked network structure.[14]

When the TGA curve of pure Elvaloy 1330 was compared with those of the samples irradiated at different dosages of irradiation, it was quite apparent that the onset of degradation of the treated samples increased progressively with increasing treatment level, although all of the samples, including the base reference compound, were degraded completely within the range of temperature under study. However, the irradiated samples possessed marginally higher onset temperatures of degradation than the pure compound.

It is quite interesting to note that the temperature corresponding to the peak rate of degradation, as evidenced from the differential thermogravimetry curves, underwent an increase from the untreated pure compound to the sample treated with 6 Mrad of irradiation, beyond which there was a marginal decrease. This may have been due to molecular chain scission, which came into play at a higher crosslink density.

## DSC

The DSC studies based on the series of constant 1-phr TMPTMA incorporation and different dosages of radiation are shown in Figure 8(a,b), respectively. The curves represent changes in the α-transition zones from 0 to 15 Mrad doses of irradiation. A marked rise in the α-transition temperature is relevant when higher irradiation dosages are incident in the system.[15] The α transition associated with the pure Elvaloy copolymer, which was observed at −125°C, was greatly modified in the presence of sensitizer under the influence of electron-beam irradiation. As expected, with an increase in the crosslink density, the segmental motion ceased, as a result of which the α transition shifted to −104°C in D112. As the crosslink density of the system increased, the energy required for the movement of the main chains also increased. This may have been due to the increasing formation of crosslinks within the

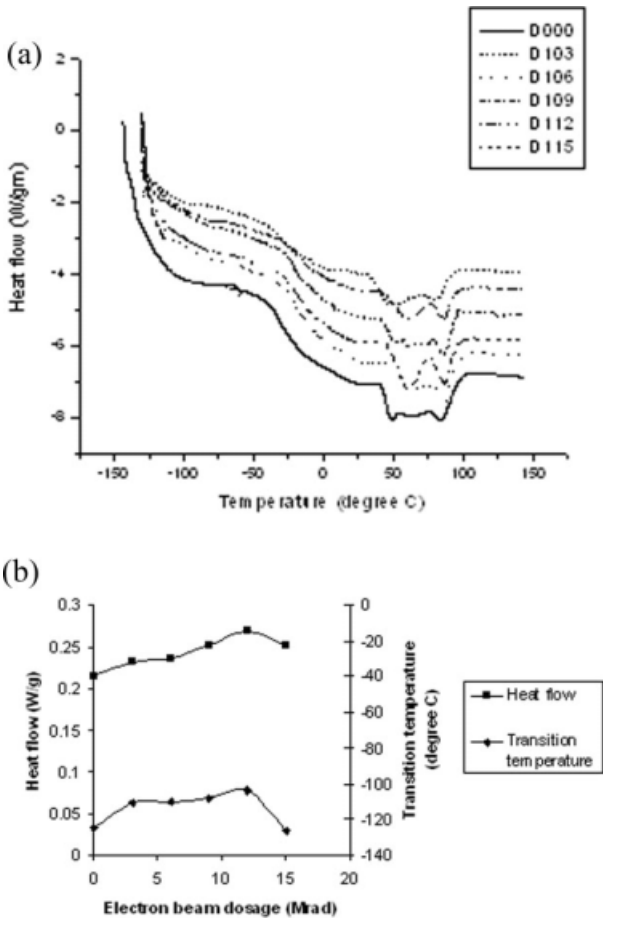



**Figure 8** (a) DSC curves at 1-phr TMPTMA incorporation. (b) Variation of the α transition temperature and energy absorbed with the electron-beam dosage.

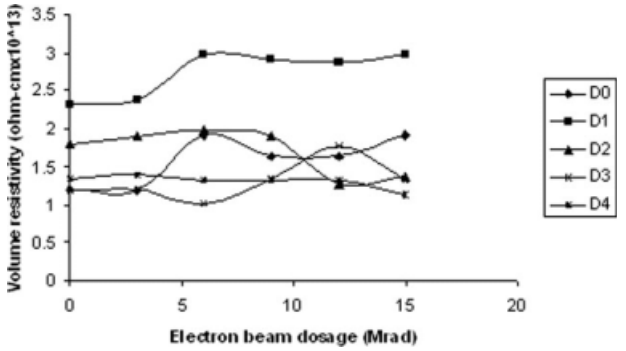



**Figure 9** Variation of the volume resistivity with the electron-beam dosage.

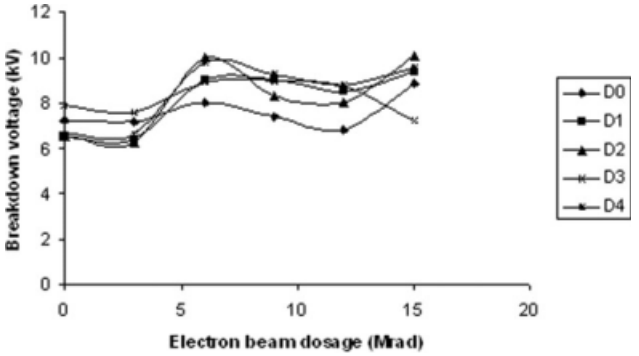



**Figure 11** Variation of the breakdown voltage with the electron-beam dosage.

main chains of the system, which imparted a compact crosslinked network structure and thus increased the energy needed at the transition zones. As a result, although the glass-transition temperatures remained almost invariant with increasing electron-beam dosage, the energy required for $\alpha$ transition gradually increased.

However, at the higher level of irradiation, the decrosslinking and chain scission processes were initiated, and the ultimate products remained in dynamic equilibrium with the formation of crosslinked products.

### Electrical properties

The electrical properties of polymers cover an extremely diverse range of molecular phenomena. The volume resistivity, dielectric constant, and breakdown voltage of the ethylene–methyl acrylate samples were measured at a constant TMPTMA level with variation of electron-beam dosage and also at various concentrations of TMPTMA at a constant irradiation dose, as shown in Figures 9–11.

The volume resistivity depends on the presence of free charges (electrons or ions) and their mobility. Here, it remained almost invariant with changing radiation dosage. This was probably because of the balancing of chain scission against that of crosslink density. It was quite obvious that with increasing crosslink density in the somewhat polar copolymer, the movement of the dipoles were increasingly inhibited by the increase in viscosity, as could be anticipated from the increasing gel content with increasing irradiation dose. However, the increasing proportion of chain scission at higher levels of sensitizer incorporation, manifested by a reduction in the mechanical properties, led to a decrease in viscosity and thus the maintenance of a level of constancy in the volume resistivity. At lower levels of sensitizer and in the sample without any sensitizer, the volume resistivity showed an increasing tendency, only because of the restricted orientation of the dipoles with increasing crosslink density.

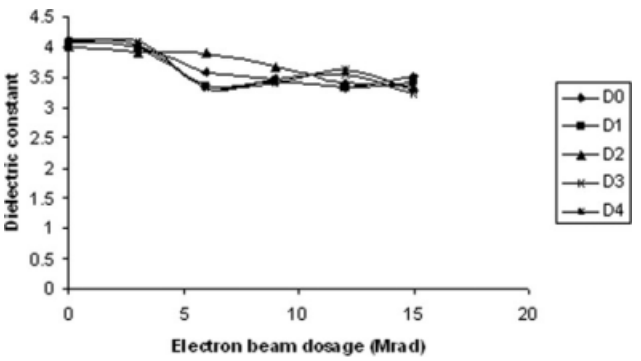



**Figure 10** Variation of the dielectric constant with the electron-beam dosage.

The dielectric constant of a polymer arises from the various molecular phenomena that come into play when the polymer is subjected to an electric field. The orientation polarization contributes a major part of the total polarization for polar polymers such as ethylene–methyl acrylate copolymers. It arises because of the presence of dipoles associated with the chains, either due to chemical construction of the material or other effects such as oxidation or diffusion of polar molecules from the environment.[16] The dipolar polarization of such molecules depends on the segmental mobility. As there are several types of motion in polymer molecules, including the main chain and the side groups, it is to be expected that polymer molecules have many relaxation temperatures that can be assigned to the motion of different parts of the molecule. The higher the viscosity of the medium (polymer matrix) is, the greater will be the hindrance to free dipolar orientation polarization from the neighboring molecules and the lower will be the dielectric constant.[17]

The dielectric constant or permittivity gives us the ratio of an electric capacitor filled with the polymeric substance under study to that of the same capacitor *in vacuo* at a definite field frequency. In this case, the dielectric constant almost exhibited constancy to a

very slow marginal decrease with increasing irradiation dosage in the system for all dosages of sensitizer under study. With increasing dosage level of irradiation, the increasing crosslink density raised the viscosity of the polymer matrix such that it reduced the possibility of dipolar orientation polarization from the adjacent molecules. The effect of increasing crosslink density was counterbalanced to some extent by the chain scission, which resulted in the deterioration in mechanical properties, as observed earlier. This effectively resulted in a marginal decrease in the dielectric constant, particularly at higher dosages of irradiation.

The breakdown of polymer dielectrics is closely related to the interfacial polarization within the polymer matrix. In this case, the presence of a single polymeric phase eliminated the possibility of space charge buildup across the microscopic interfaces; this thus gave almost invariant values of breakdown strength along increasing irradiation dose.[18]

## CONCLUSIONS

The effect of electron-beam irradiation on Elvaloy 1330 at various levels of the sensitizer TMPTMA were evaluated. The tensile strength increased gradually at lower irradiation dosages but showed a gradual decrease after an optimum value in each case was reached. The samples with higher sensitizer incorporation optimized at lower radiation doses compared to the ones with the incorporation of lower levels of TMPTMA. The elongation at break and tear strength showed a constant fall and were directly proportional to each other, whereas the modulus and hardness displayed a steady increase with variation in irradiation dose level. The electrical properties remained almost invariant with changes in irradiation dose, whereas the thermal characteristics were well consistent with the changes in crosslink density.

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